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*Molybdenum - Corrosion · Hi Temp.*

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To:             DEPARTMENT OF THE NAVY
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On:             PROTECTION OF MOLYBDENUM AGAINST
                CORROSION AT HIGH TEMPERATURES

For the period: April 1, 1954 - September 31, 1954

Submitted by:   J. W. Spretnak and Rudolph Speiser
                Department of Metallurgy

Date:           September 9, 1954

# PROTECTION OF MOYLBDENUM AGAINST CORROSION AT HIGH TEMPERATURES

## I. INTRODUCTION

Work during the past six months has been chiefly directed towards producing molybdenum-nickel and molybdenum-cobalt alloys, which at high temperatures in air will produce self-regenerative, protective coatings that will not spall off the surface of the alloy on cooling.

With this end in view investigation of the following has been continued:

1) The mechanism of spalling in cobalt molybdate.

2) The mechanism of spalling in nickel molybdate.

3) The prevention of spalling in cobalt and nickel molybdates.

4) Testing of Mo-Co-X and Mo-Ni-X alloys.

As a preliminary to an investigation of the Mo-Zr and Mo-Cu alloys, zirconium molybdate and copper molybdate have been prepared and their X-ray diffraction patterns obtained.

## II. EXPERIMENTAL WORK

### A. MECHANISM OF SPALLING IN COBALT MOLYBDATE

Further differential thermal analyses have now been carried out very carefully on different samples of cobalt molybdate. The results throw considerable illumination on the mechanism of spalling.
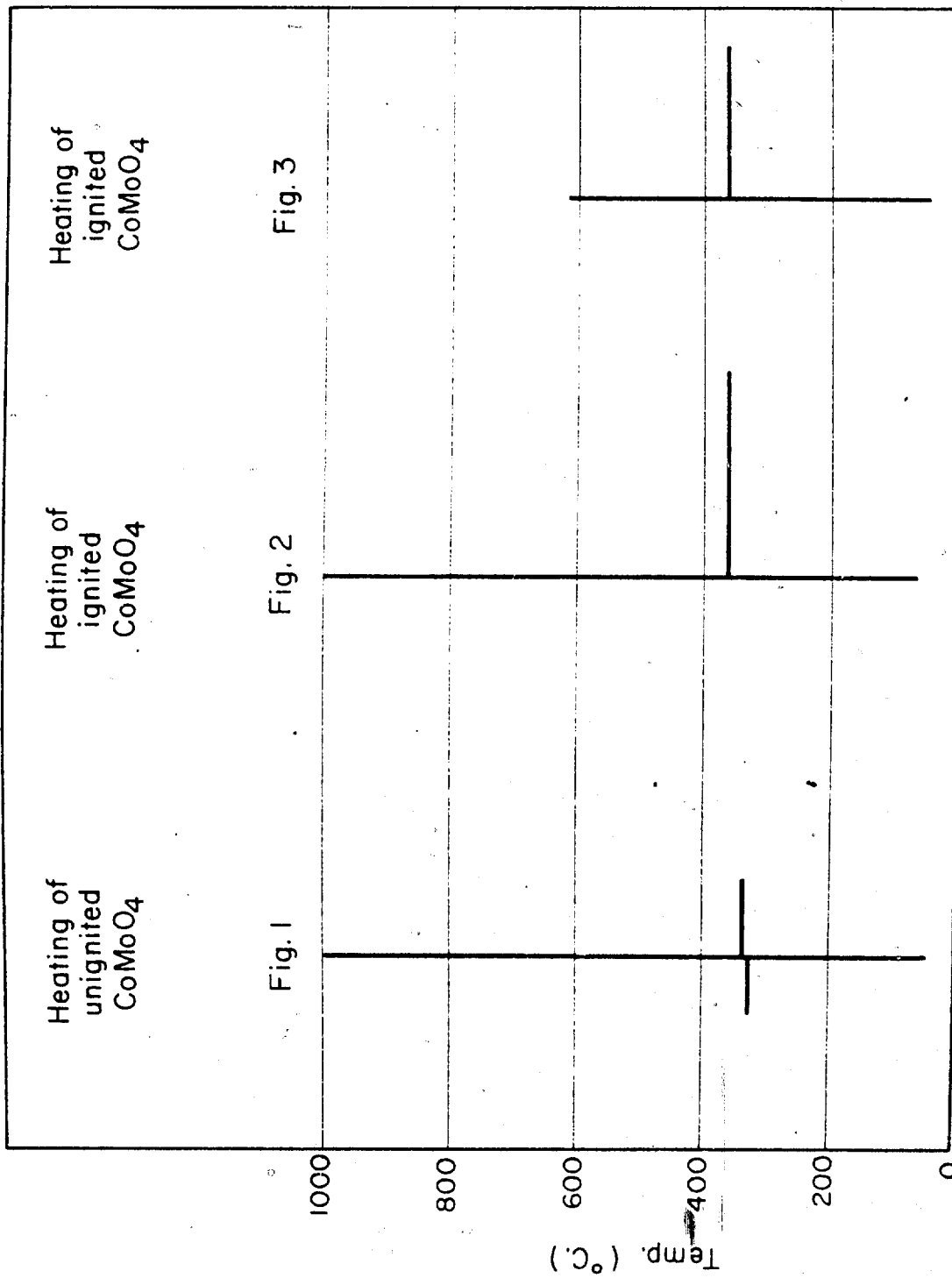
The technique consists in heating under the same conditions the sample under test and a substance which has no polymorphic changes.

The temperature difference between the two is recorded. Where no change in phase of the sample occurs the temperature difference is approximately zero. At a phase change, the evolution or absorption of heat in the sample causes an abrupt change in the temperature difference, which is recorded by a Brown Electronik differential recorder. The technique is described in greater detail elsewhere[1,2].

The results are plotted graphically in Figures 1 to 7. The positions of the horizontal lines indicate the temperature at which the transitions occur, and the lengths of these lines as drawn are the same as those of the deflections shown on the differential thermogram. The horizontal lines to the left indicate exothermic transitions; those to the right, endothermic transitions.

Figure 1 shows the results of differential thermal analysis on a sample of unignited cobalt molybdate; that is, the room-temperature phase of cobalt molybdate. Two transitions occur, one exothermic at $325°$ and one endothermic at $336°C$. Another endothermic inflection appears at $922°C$. It seems to be reversible on cooling. However it is so small that it is not certain that it corresponds to a phase change. It has therefore not been included in the figures, in order to avoid further complication. It has previously been shown[1] that neither on cooling nor on reheating do either of the transitions at $325°C$ or $336°C$ reappear.

Figure 2 shows the differential thermal analysis of a sample of cobalt molybdate which had been previously ignited for a long time and cooled. An X-ray diffraction pattern showed this sample

Heating of unignited CoMoO₄ — Fig. I

Heating of ignited CoMoO₄ — Fig. 2

Heating of ignited CoMoO₄ — Fig. 3

Temp. (°C.)

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to consist purely of the high-temperature phase of cobalt molybdate[3].

On heating this sample only the endothermic transition at about 358°C

occurs. Neither on cooling nor on reheating does this transition

reappear. If, however, the sample is afterwards cooled in snow

carbon dioxide then on reheating the endothermic transition at 358°C

is recovered (Figure 3). An X-ray diffraction pattern of the sub-

sequently cooled sample showed it to consist of a mixture of the high-

temperature and intermediate-temperature phases[3]. Further cooling

with snow carbon-dioxide and with liquid nitrogen results in progres-

sive increases in the proportion of the high-temperature phase and

less of the intermediate-temperature phase as shown by X-ray diffrac-

tion patterns.

It was also observed that the intermediate-temperature phase

could be completely changed to the high-temperature phase simply by

the action of mechanical grinding at room temperature.

It had been previously noted[3] that:

(a) Spalling occurs almost at room temperature.

(b) When spalling occurs the product is always pre-
dominantly the high-temperature phase.

(c) When spalling does not occur after cooling the
product is always predominantly the intermediate-
temperature phase.

(d) Only when cobalt molybdate has been heated to a
high-temperature (above about 700°C) for a suf-
ficiently long time does spalling occur with
formation of the high-temperature phase.

(e) The room-temperature phase has never been re-
covered after cobalt molybdate has been heated
above 325°C.

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The following conclusions are therefore reached:

1) On heating the room-temperature phase of cobalt molybdate an exothermic phase change occurs at $325^{\circ}C$ and the high-temperature phase results.

2) The high-temperature phase is stable only on heating up to about $350^{\circ}C$. Further heating produces an endothermic change and results in a third phase, the intermediate-temperature form. The terms "high-temperature" and "intermediate-temperature phases" are therefore really misnomers. In the future the forms known previously as the "room-temperature," "high-temperature," and "intermediate-temperature" phases will be replaced respectively by the terms $\alpha$, $\beta$ and $\gamma$ phases.

3) On cooling, the transition from the $\gamma$ to the $\beta$ phase occurs not at about $350^{\circ}C$ as might be expected, but very near room temperature.

4) The phenomenon of spalling is "structural"[4]. It consists in this transition from the $\gamma$ to the $\beta$ phase on cooling.

5) The exact temperature of the above transition depends on the previous history of heating of the specimen. The reason for this is not yet understood.

B. MECHANISM OF SPALLING IN NICKEL MOLYBDATE

The differential thermal analysis of unignited nickel molybdate (the room-temperature form) was repeated (Figure 4). Contrary to the results previously obtained[1] only one transition (exothermic, at $418^{\circ}C$) was observed, instead of two. The experiment was repeated at a different rate of heating, but sill only one inflection was obtained. It is believed that the second inflection, which was previously found and which is to be expected by analogy with cobalt molybdate, must be inhibited for some reason in this particular

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Heating of unignited NiMoO₄

Fig.4

Cooling of NiMoO₄

Fig.5

Heating of ignited NiMoO₄

Fig.6

Cooling of NiMoO₄

Fig. 7

Temp. (°C.)
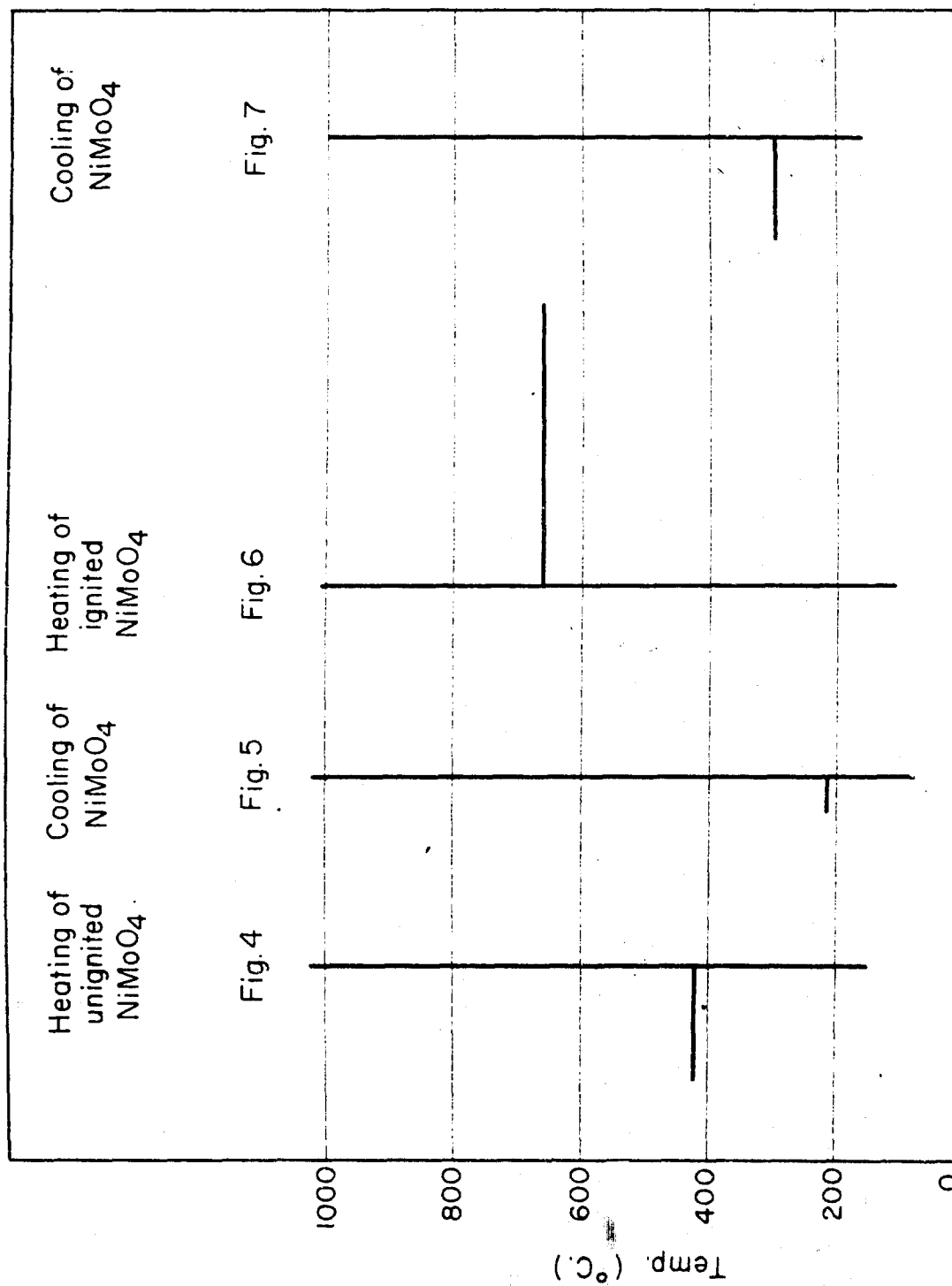
sample, so that only a fraction of the sample undergoes this phase change. The heat of transition therefore is small and was not detected on the thermogram. On cooling (Figure 5) an exothermic change occurs at $214^\circ C$. It is believed that this corresponds to the endothermic change which was not recorded on the thermogram on heating.

It is interesting to note that in the differential thermal analysis of nickel molybdate presented in a previous report[1] a series of exothermic changes were exhibited on cooling. It is known, however, that a single transformation can sometimes occur in a series of bursts over a wide range of temperature[5]. The nonreproducibility of the results can be rationalized by postulating the importance of grain size as a factor in phase transformations.

It is believed that, as with cobalt molybdate, the terms "high-temperature" and "intermediate-temperature phase" used in previous reports are misnomers and here as well in future the forms known previously as the "room-temperature," "high-temperature," and "intermediate-temperature" phases will be replaced respectively by the terms $\alpha$, $\beta$ and $\gamma$ phases.

It is thought that, by analogy with cobalt molybdate, this exothermic change on cooling is due to the transformation from the $\gamma$ phase to the $\beta$ phase; and that spalling is associated with this phase change. The final product, as might be expected from the work performed on cobalt molybdate, was a mixture of the $\beta$ and $\gamma$ phases of nickel molybdate, with the $\beta$ phase strongly predominating.

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The results of further experiments tend to confirm the above ideas.

For example, differential thermal analysis was performed on a sample of nickel molybdate synthesized by heating stoichiometric proportions of molybdenum trioxide and nickel oxide (NiO) under vacuum in a sealed Vycor tube at about $900^\circ$C for several days. This sample consisted of the $\beta$ phase only of nickel molybdate. The results of the thermal analysis are presented in Figure 6. The exothermic transition at $418^\circ$C from the $\alpha$ to the next phase on heating does not, of course, occur. But the predicted endothermic transition is recorded very distinctly at $657^\circ$C. On cooling, this phase change reverses at $296^\circ$C and the $\beta$ phase reforms.

Differential thermal analyses of samples of nickel molybdate which have been stabilized in the $\gamma$ phase by heating with equimolecular proportions of manganese dioxide show no transitions either on heating or cooling.

## C. PREVENTION OF SPALLING IN COBALT AND NICKEL MOLYBDATE

Work was continued on those systems in which the elimination of spalling in the molybdate has already been observed. Some new systems were also introduced.

It is known that $ZnWO_4$ has a stable crystal structure of a type possibly related to some forms of nickel and cobalt molybdates. It was thought that zinc might be a possible stabilizing additive to the nickel and cobalt molybdates, transforming them into the stable $ZnWO_4$ structure.

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A similar consideration suggests that copper may combine with molybdenum and oxygen to yield the stable $Ag_2MoO_4$ type structure.

(a) $MnO_2$-$NiMoO_4$ system

Nickel molybdate and manganese dioxide were heated together for periods of about one day at about $1100^\circ C$ in the following proportions: 1:1/4, 1:1/2, 1:1, 1:2, 1:4. In those mixtures where the proportions were from 1:1/2 to 1:2 the structure resulting was isomorphous with the $\gamma$ phase of cobalt molybdate. It was presumed to be the stabilized $\gamma$ form of nickel molybdate not previously isolated. The d spacings of the structure increased as the proportion of manganese dioxide was increased. In the sample where the proportion of nickel molybdate to manganese dioxide was only 1:1/4 both the $\beta$ and $\gamma$ phases of nickel molybdate were found. In the sample where the proportion reached 1:4 another, unidentified structure formed.

(b) $MnO_2$-$CoMoO_4$ system

Equimolecular quantities of manganese dioxide and cobalt molybdate heated at $980^\circ C$ for twenty-two hours resulted in complete stabilization of cobalt molybdate in the $\gamma$ phase on cooling.

(c) $Al_2O_3$-$CoMoO_4$ system

A more extensive investigation of this system led to the following conclusions.

The reaction between alumina and cobalt molybdate, which results in stabilization of cobalt molybdate in the $\gamma$ phase, is slow. Moreover the cobalt molybdate was never completely stabilized in the experiments performed. Further experiments performed over more

prolonged periods at higher temperatures indicated that the final

product obtained was not the stabilized $\gamma$ phase which is apparently

only an intermediate stage, but the spinel $CoO \cdot Al_2O_3$.

(d) $SiO_2-CoMoO_4$ system

A preliminary investigation of this system shows that silica

can stabilize cobalt molybdate, although in the experiments performed

stabilization was never complete. An unidentified structure also

forms and it is possible that this may be the end product of the

reaction. However, insufficient results have been collected to permit

even tentative conclusions to be drawn.

(e) $ZnO-NiMoO_4$ system

Equimolecular proportions of zinc oxide and nickel molybdate

heated at $1112^{o}C$ for 44 hours did not result in the formation of even

traces of the stabilized $\gamma$ phase.

(f) $ZnO-CoMoO_4$ system

Equimolecular proportions of zinc oxide and cobalt molybdate

heated at $1112^{o}C$ for 44 hours resulted in partial but not complete

stabilization of cobalt molybdate.

(g) $CuO-NiMoO_4$ system

Equimolecular proportions of cupric oxide and nickel molybdate

heated at $980^{o}C$ for 16 hours gave a compound which appeared to have

melted and which spalled violently on cooling.

(h) $CuO-CoMoO_4$ system

Heating equimolecular proportions of cupric oxide and cobalt

molybdate at $980^{o}C$ for 16 hours led to the same observation as was made

on the $CuO-NiMoO_4$ system.

D. OXIDATION TESTS ON ALLOYS

The apparatus and technique used in the oxidation tests were described in previous reports[1,6]. The technique permits weighing of the oxidizing sample at any time during the test.

The alloys chosen were those, which, from the results obtained in the stabilization of cobalt and nickel molybdates, and from previous oxidation tests, appeared likely to produce completely protective, stabilized coatings.

1) Oxidation of the alloy 68% Mo - 22% Ni - 10% Mn.

An alloy of this composition was chosen because:

(a) Calculation based on an estimate of the density
of nickel molybdate showed that in this alloy
the nickel molybdate formed during oxidation
should completely cover the surface of the alloy.

(b) The percentages of nickel and manganese in the
alloy are in the molecular ratios 1:1/2. This
proportion of manganese was shown previously in
the report to be sufficient to result in complete
stabilization of nickel molybdate.

The alloy was synthesized by sintering a compact of the metal powders in hydrogen for 90 hours at 1130°C.

The results of the oxidation test are shown in Figures 8 and 9.

Figure 8 represents the oxidation of the alloy at 940°C for a period of 22 hours. On removal from the furnace some powder spalled off the sample. An X-ray diffraction pattern showed this powder to be a mixture of the $\beta$ and $\gamma$ phases of nickel molybdate.

The specimen was replaced in the furnace and a more prolonged oxidation test performed (Figure 9). The specimen seems to be
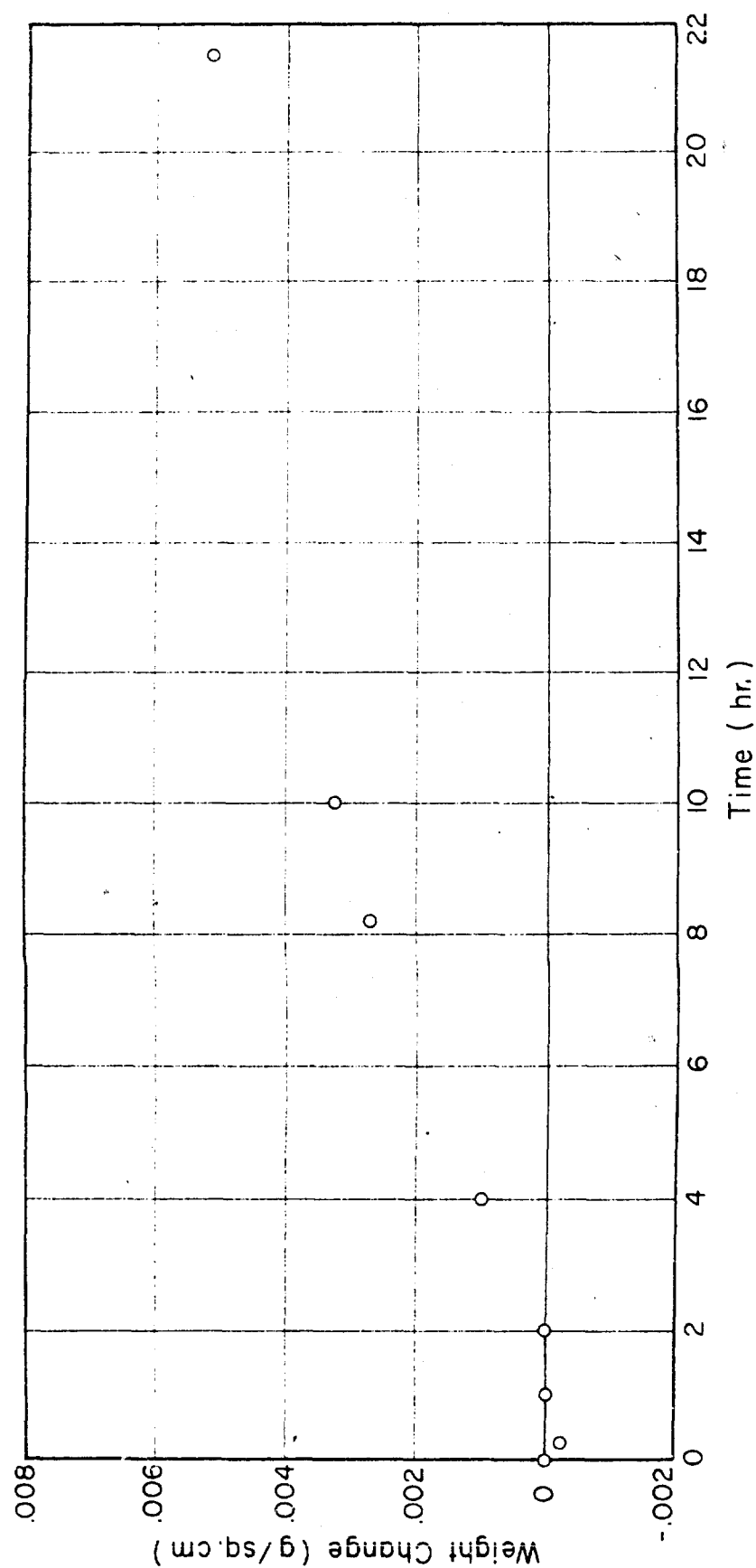
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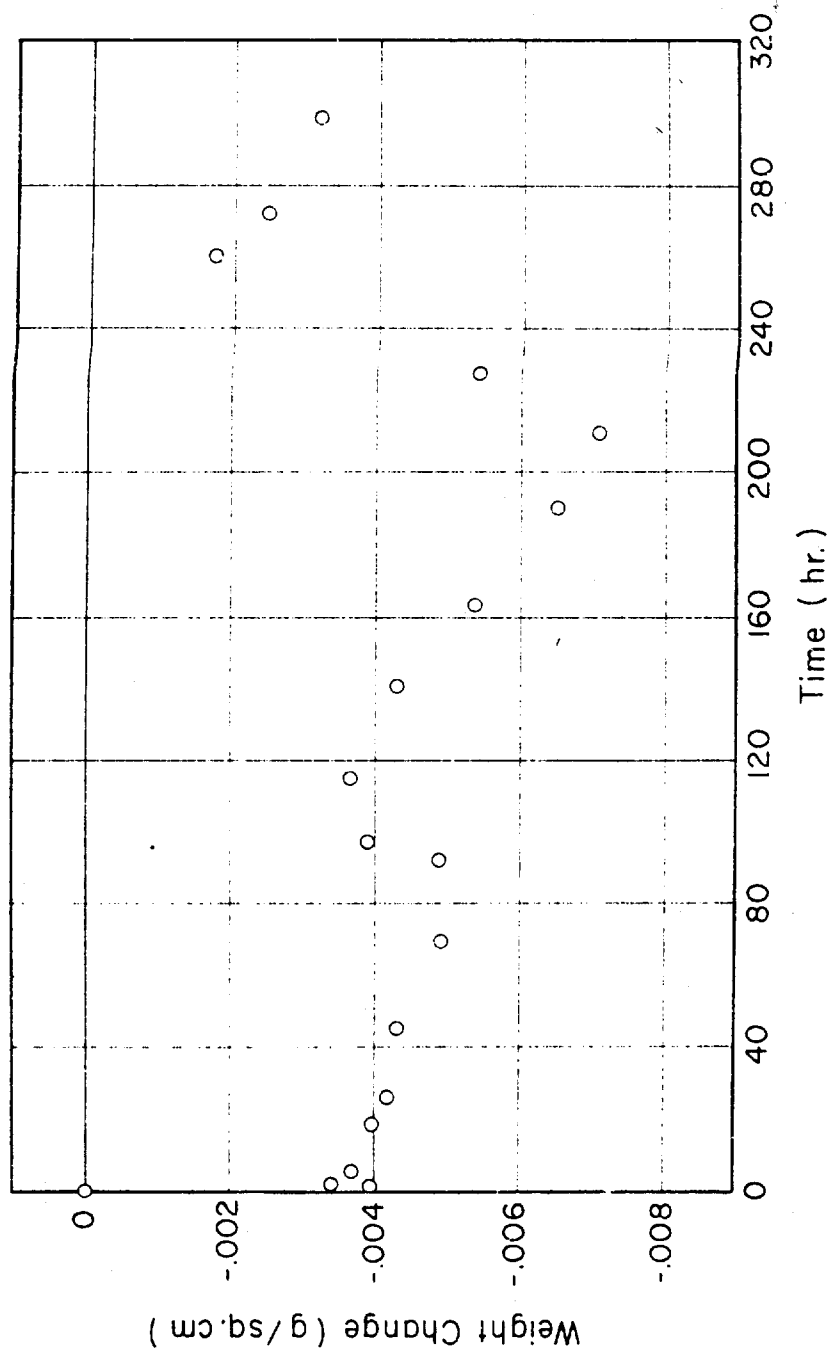Fig.8. Oxidation Curve for Alloy 68% Mo – 22% Ni – 10% Mn. Test Temp.– 940° C.

Fig. 9. Oxidation Curve for Alloy 68% Mo − 22% Ni − 10% Mn.
Test Temp. − 940° C.

gaining and losing weight in cycles. It has not been considered justifiable to join the points on these graphs. On cooling the specimen a green powder spalled off the surface and was again identified as consisting predominantly of the β and γ phases of nickel molybdate.

It seems that on the surface of this alloy the nickel molybdate was partially but not completely stabilized.

2) Oxidation of the alloy 68% Mo - 22% Co - 10% Mn.

This alloy corresponds to the Mo-Ni-Mn alloy described in the previous section. It was synthesized in the same way.

Its oxidation curve is presented in Figure 10. The specimen increases in weight fairly rapidly. On removal from the furnace only a very tiny amount of the surface coating spalled. An X-ray diffraction pattern showed that the β phase of cobalt molybdate was absent in the coating. The stabilized γ phase was present and its d spacings were slightly larger than in pure cobalt molybdate, indicating that manganese had played a part in the stabilization. Some unidentified lines were also present in the pattern.

3) Oxidation of the alloy 63% Mo - 25% Co - 12% Si.

Prolonged testing of this alloy led to disappointing but interesting results. It was shown in the previous report[4] that this alloy produces an almost completely protective coating of stabilized cobalt molybdate on heating for 160 hours at 960°C. Further heating, however, results in an abrupt decrease in protectivity (AB, Figure 11). On removal of the specimen from the furnace after 340 hours the coating
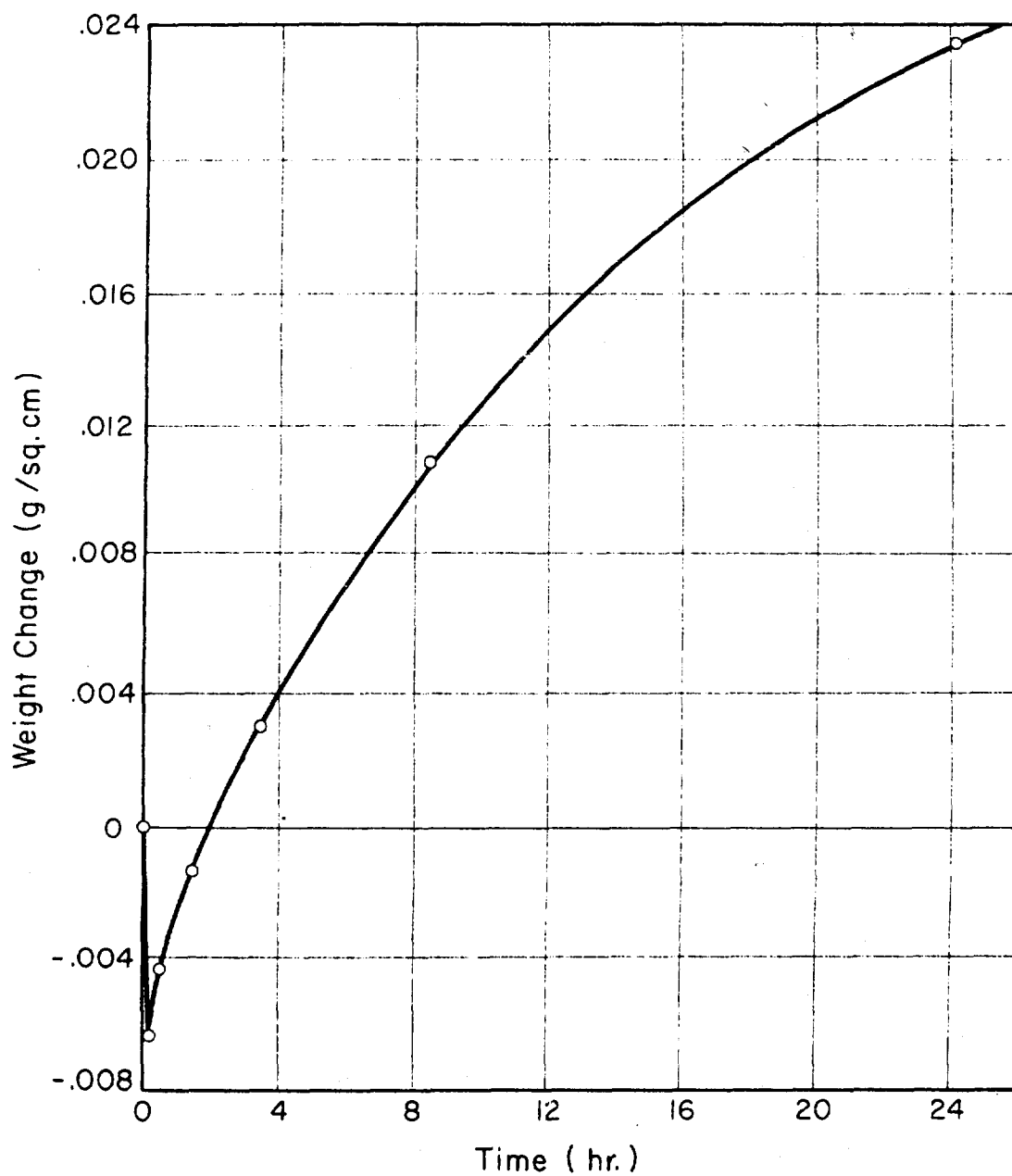
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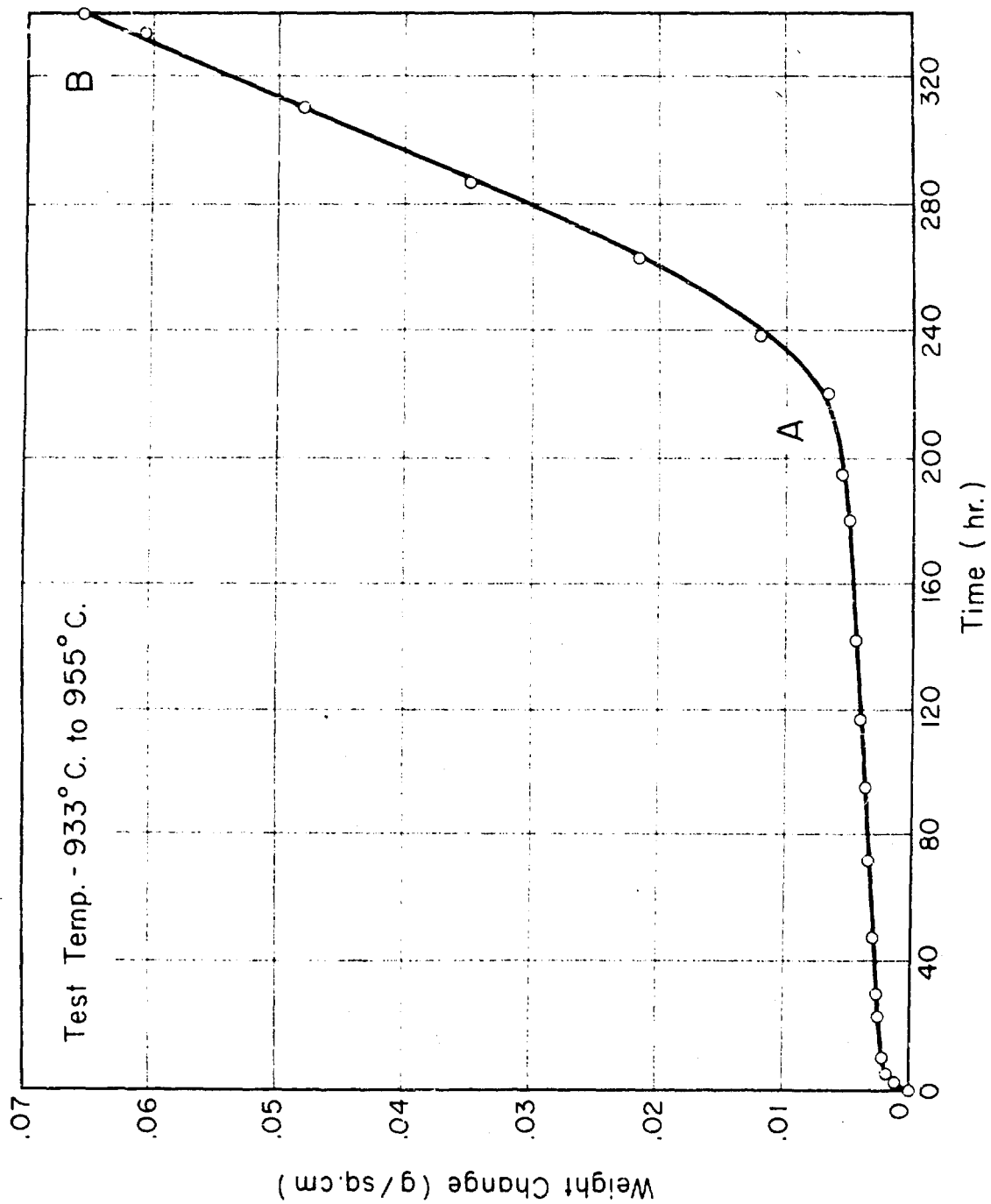Fig. 10. Oxidation Curve for Alloy 68% Mo – 22% Co – 10% Mn.
Test Temp. – 940°C.

Fig. II. Oxidation Curve for Alloy 63% Mo – 25% Si – 12% Co.

Test Temp. - 933° C. to 955° C.

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spalls violently off the alloy. An X-ray diffraction pattern showed this coating to consist of the β phase of cobalt molybdate and another unidentified substance. The stabilized γ phase of cobalt molybdate was absent.

## E. SYNTHESIS OF ZIRCONIUM MOLYBDATE $Zr(MoO_4)_2$ AND CUPRIC MOLYBDATE $CuMoO_4$

### 1) Synthesis of zirconium molybdate.

Zirconium dioxide and molybdenum trioxide in the molecular ratios 1:2 were heated together in a sealed Vycor tube under vacuum for 3 days at $830^oC$ and two more days at $910^oC$. A discolored white powder was formed. An X-ray diffraction pattern indicated that the zirconium dioxide and molybdenum trioxide had reacted completely. A preliminary examination of the pattern indicated that the zirconium molybdate product may have a crystal structure which is only a slight distortion of a more symmetric lattice. Identification of lattice type has not been completed.

### 2) Synthesis of cupric molybdate.

Stoichiometric proportions of cupric oxide (CuO) and molybdenum trioxide were heated in a sealed Vycor tube under vacuum for 2 days at $830^oC$. On removal from the furnace it was noted that the specimen had melted. It consisted predominantly of a green substance with traces of a brown substance present.

## III. SUMMARY

Spalling in cobalt molybdate on cooling is "structural." It is due to an exothermic phase change from the γ phase to the β phase on

cooling. It appears extremely likely that spalling in nickel molybdate is almost exactly analogous to that in cobalt molybdate.

Stabilization in the $\gamma$ phase can be effected in nickel molybdate by manganese dioxide. In cobalt molybdate it can be effected by manganese dioxide and possibly by silica or zinc oxide, although it is not yet known whether these last two can produce a complete or permanent stabilization. The $\gamma$ phase of cobalt molybdate is stabilized by alumina only as a step in the production of the spinel, $CoO \cdot Al_2O_3$. Further work on the alumina - cobalt molybdate system is therefore being deferred until there is time for an investigation on the possibility of protecting molybdenum with spinel coatings.

Results on Mo-Ni-Mn and Mo-Co-Mn alloys show promise. A partially stabilized coating of nickel molybdate was formed on the alloy 68% Mo - 22% Ni - 10% Mn. An almost completely stabilized coating of cobalt molybdate was produced on the alloy 68% Mo - 22% Co - 10% Mn. Neither of these coatings was completely protective but it is believed that adjustments of the percentages of the constituents in these alloys will result in the formation of stabilized completely protective coatings.

Oxidation of the alloy 63% Mo - 25% Co - 12% Si indicates that stabilized cobalt molybdate forms on the surface, but only temporarily. The reactions at the surface are not yet properly understood.

Zirconium molybdate and cupric molybdate have been prepared. Cupric molybdate has a melting point below $830^\circ C$, so further work on it will be dropped.

# IV. FUTURE WORK

It is expected that a high-temperature X-ray camera will soon become available. It will be used in an attempt to confirm the theory of spalling in cobalt and nickel molybdates, to obtain the crystalline structures of the various phases, and to find if there are any further phase transformations that were missed during differential thermal analysis.

The manganese dioxide - nickel molybdate and manganese dioxide - cobalt molybdate systems will be further investigated in an attempt to find how manganese enters the molybdate lattice and how stabilization results.

The systems silica-cobalt molybdate and zinc oxide-cobalt molybdate will be further investigated to see if permanent, complete stabilization of cobalt molybdate can result in either system.

Oxidation tests will be continued on Mo-Ni-Mn and Mo-Co-Mn alloys of varying compositions designed to produce protective, stabilized molybdate coatings.

Differential thermal analysis and volatility measurements will be made on zirconium molybdate in order to see if this compound has any possibility of forming a protective coating on molybdenum.

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